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CENTRAL FAX CENTERNalco Docket No. 7774  
Customer No. 49459

MAY 19 2009

In the United States Patent and Trademark Office

Applicants:	Brian T. Holland et al.	)	Examiner:	Daniel S. Metzmaier
		)		
Serial No.:	10/827,214	)	Art Unit:	1796
		)		
Date Filed:	April 19, 2004	)		

For: COLLOIDAL COMPOSITIONS AND METHODS OF PREPARING SAME

Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450**DECLARATION OF PRIOR INVENTION IN THE UNITED STATES OR IN A NAFTA OR  
WTO MEMBER COUNTRY TO OVERCOME CITED PATENT OR PUBLICATION  
UNDER 37 C.F.R. § 1.131**

Dear Sir:

This Declaration is to establish completion of the invention in this application in the United States prior to December 5, 2003, the effective date of Cundy et al., "Some observations on the preparation and properties of colloidal silicates. Part I: synthesis of colloidal silicalite-1 and titanasilicalite-1 (TS-1)," Microporous and Mesoporous Materials, 66 (2003): 143 to 156 ("Effective Date of Cundy").

To establish a date of completion of this invention prior to the Effective Date of Cundy, Applicants submit herewith copies of research notebook pages attached as Exhibit A hereto. This exhibit clearly and definitely establishes invention of the subject matter of the currently rejected claims prior to the Effective Date of Cundy.

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Applicants declare that the document submitted herewith as Exhibit A is dated prior to December 5, 2003. Accordingly, Applicants respectfully assert that this invention was completed prior to the Effective Date of Cundy.

**DECLARATION**

As a person signing below, I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: 5/19/09

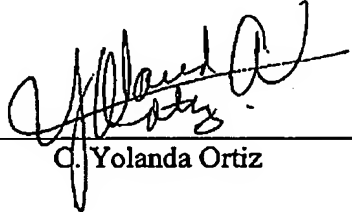
Signed:   
C. Yolanda Ortiz

Exhibit A

Page 1 of 3

TITLE  $\text{Ce}_2\text{O}_3/\text{SiO}_2$ 

PROJECT NO.

BOOK NO.

3

Preparation of  $\text{Ce}_2\text{O}_3\text{-SiO}_2$  Co-sol

$\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$   $\rightarrow$   $\text{Ce}_2\text{O}_3$   $\frac{\text{Ce}_2\text{O}_3}{2 \text{ Ce}(\text{NO}_3)_3} = \frac{328.24}{868.44} = 377.9$

For 5%  $\text{Ce}_2\text{O}_3$  soln,  $5/377.9 = 13.23 \text{ g}$  of nitrate

$\text{NO}_3$   $\frac{184}{434.23} = 3 \text{ mg}$   $\frac{0.0069 \text{ mg}}{100 \text{ g}} = 0.0069 \text{ mg/g}$   
 $\text{Ce}(\text{NO}_3)_2$   $\frac{434.23}{434.23} = 1$   $\frac{0.0010 \text{ mg}}{100 \text{ g}} = 0.0010 \text{ mg/g}$   
 $= 0.438 \times 13.23 \text{ g} = 5.80 \text{ g NO}_3$   $\frac{5.80 \text{ g}}{100 \text{ g}} = 5.80 \text{ g}$   $\frac{1}{100 \text{ g}} = 5.80 \text{ g}$

11g KOH + 200ml DIW pH 13.34  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   
 AS 1.12 11kg pH = 9.05  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   
 SA 201.042 6.88  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$

E	W	E/W	pH	T
0	0		10.8	76
9:30	33.7	99.2	7.4	74
2:01	95	9.26	7.4	74
1:02	22.3	8.23	7.3	73
1:15	260	7.9		
1:29	322	8.21	7.3	73
1:53	411	7.79	7.5	75
2:09	467	7.85	9	90
2:27	535	7.99		123
3:10	689	7.35		5
3:24	738	7.40		20
3:45	816	7.44		75
4:02	875	7.60		93
4:10	906	7.80		105
4:57	1075	7.80		125

heated an extra hour

2/6 pH = 7.30 T = 19 C  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ pH = 7.90 C = 9.40  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ 

100 ml left del. next hour

2/7 1st. Filtered 1.25  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ 2nd C = 4.40  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ 3rd C = 2.85  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ Can concentrate  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ pH = 7.45 C = 4.35  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ Qualytest: Filtered 25 ppm  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ Na 86 ppm K 890 ppm  $\text{NO}_3 < 1.9 \text{ ppm}$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ SIGNATURE  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ DATE  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ READ AND UNDERSTOOD BY  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$ DATE  $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$   $\frac{1}{19.5} = 2.86$

Exhibit A  
Page 2 of 3

TITLE  $\text{SiO}_2/\text{TiO}_2$   
Co - Sol

PROJECT NO.  
BOOK NO.

17

Reaction with  $\text{TiOSO}_4$

AS = 1100g  $\text{C} = 215 \text{ ml}$   $\text{pH} = 2.32$   
 Sp Gr 1.04 6.56%  $\text{SiO}_2$   
 1100g 6.56% = 72.48g  $\text{SiO}_2$   $\times 2 \text{ AT } \text{TiO}_2 = 1.44 \text{g } \text{TiO}_2$   
 $\Rightarrow 3.3 \text{g } \text{TiOSO}_4 \cdot x \text{H}_2\text{O}$  from GFS 150g 3rd  
 added to AS  $\Rightarrow \text{pH} = 1.70$   $\text{C} = 750$   $\text{Ti} = 863 \text{ mg}$

Next  
 0.75 KOH 1 pH = 1.70  $\text{C} = 750$   $\text{Ti} = 863 \text{ mg}$   
 2nd add 2nd 12.47 g  $\text{Ti} = 907 \text{ mg}$

t	vol	pH	T	Notes
0	0	1.70	770	3.66 ml/min
10	37	9.55	72	
20	32	8.90	72	
32	110	7.96	73	addition of 0.1N KOH starts
41	151	7.20		20 ml added
1:26	241	6.34		40
1:23	267	5.84	73	50
1:25	312	6.2		100
1:50	402	6.8		185
2:13	485	7.12	74	350
2:25	531	6.79		250 ml more in the front
2:40	589	6.42	74	15
3:03	671	6.80		100
4:00	878	6.67	72	225
4:38	1021	6.17	74	250
5:15		6.24		stop
3:15		7.10	17	Refined for 1-hr

Very thick, no ppt and gel  
 pH 6.5  $\text{C} = 1310$   
 U.F. starts

3/28 filters very slow  
 1st filtrate 550 ml pH = 5.60  $\text{C} = 1350$   
 500 ml added to the cell  
 2nd filtrate 930 ml pH = 5.76  $\text{C} = 950$   
 4/15  
 3rd filtrate  
 710 ml pH = 6.15  $\text{C} = 520$

Continued pg 18

SIGNATURE

DATE

READ AND UNDERSTOOD BY

DATE

cep

Cheryl Sloboski

Exhibit A  
Page 3 of 3

18

TITLE

PROJECT NO.

BOOK NO.

Concentrate

Very thick, but fluid opaque

300 ml 3 of 8 C=6.55

set in test tube @

40°C w/ 18A

A

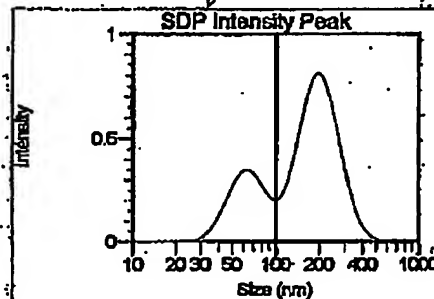
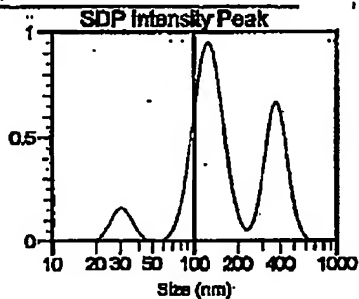
Another test tube adjusted to pH = 10.3 w/ 18A

B

After overnight @ 60°C, samples look like gel  
but re-disperse when shaken  
w/ 18A. re-disperse B. re-disperse both re-disperse w/ shaking

DELS data  
114.2 ± 49 nm  
poly 0.701

132.3 ± 56.8 nm  
poly 0.701



A Concentrate

B Dilute pH adjusted

Qualifier Report

	SiO <sub>2</sub>	Na	K	SO <sub>4</sub>
1st fill	250	6.4	440	92.37
2nd	200	4.6	300	430
3rd	87	2.7	150	340
4th			880	190
5th			960	18

RT samples with  
Thick, re-disperse  
w/ shaking  
3.2  
0.9  
1200 ± Packed @ 18.9-37.6/

Stable after 2 years

SIGNATURE

DATE

READ AND UNDERSTOOD BY

DATE

UP

Cheryl Slabogoshi